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## **METHOD FOR THE COMPARISON OF ADDITIVES FOR PORTLAND CEMENT CONCRETE**

**CAUTION:** Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read “**SAFETY AND HEALTH**” in Section G of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

### **A. SCOPE**

The procedure used for comparing samples of concrete additives with an infrared spectrophotometer is described in this test method.

### **B. APPARATUS**

1. Infrared spectrophotometer, with automatic recorder
2. Wig-L-Bug amalgamator, with stainless steel capsule, 12.7-mm diameter, cap, and steel balls
3. Laboratory Press Model P-21, Pasadena Hydraulics, or other press with a capacity of over 110 000 N or similar
4. Vacuum oven
5. Vacuum pump
6. Evacuatable KBr die
7. Disk holder for solid samples

### **C. SAMPLE PREPARATION**

1. Liquid Sample Preparation:
  - a. Liquid samples should not be evaporated directly in the vacuum

oven. Instead, place 1 to 3 ml of the as received sample on an aluminum disk. Then, place the aluminum dish in a conventional oven at 50 to 60°C, for an overnight period. This should remove most of the moisture in the sample.

- b. Place the aluminum dish containing the partially-dried sample in a vacuum oven at 60°C and gradually increase the vacuum to 101 kPa. Some materials may froth or foam excessively at this point, and it may be necessary to allow a small amount of air to bleed into the oven, to control the frothing and to remove any last traces of moisture or volatile material.
- c. After the sample has stabilized, close the air-bleed valve on the oven and continue drying. Generally, 3 h of drying in a vacuum oven, at 101 kPa, is sufficient.
- d. Remove the sample from the oven and carefully transfer it to an agate mortar. Grind the sample to pass a standard 180- $\mu$ m sieve, and return it to the vacuum oven at 101 kPa for approximately 1 h.

- e. Proceed as described in Part 3.

2. Solid Sample Preparation:

- a. Depending on their composition, solid samples may be handled in several ways. As an example, if the sample is a soluble resinous material, it may be possible to dissolve a portion in solvent and evaporate the solution directly on a previously prepared alkali halide disk. Water soluble or dispersed samples should be placed on disks of insoluble materials such as silver chloride.
- b. Non-soluble solids should be ground into pieces and dried overnight in an aluminum dish in the vacuum oven at 60°C and 101 kPa. They should then be treated as described in Part 3.

3. Specimen Preparation:

- a. Remove the sample from the oven. Grind 2 mg of the sample with 250 mg of Spectro-grade KBr until it forms an agate mortar. This breaks up any lumps of KBr and provides a preliminary mix to the specimen.
- b. Transfer the mixed material, plus one steel ball, to a Wig-L-Bug capsule and vibrate the sample for 30 to 60 s.
- c. Being careful that the steel ball is not also transferred, transfer the powdered specimen to the evacuable die, and follow the manufacturer's instructions to prepare a suitable disk.
- d. Thoroughly clean the die after each use. Be careful to avoid damaging the polished die faces.

**D. TEST PROCEDURE**

- 1. Place the disk in an infrared spectrophotometer and run the absorption curve.

- 2. Test results are used for comparison purposes only. Each curve is compared

with curves from samples run previously. Two materials are considered similar if all of the absorption points agree as to wave length. The magnitude of the absorption is also considered, although even with care in weighing and preparation of samples, a difference of  $\pm 10\%$  is often encountered.

**E. PRECAUTIONS**

Read the instructions carefully which accompany the evacuable die as to all correct procedures. Handle the die faces carefully so as not to mar or scratch the die surface.

**F. REPORTING RESULTS**

Report the test results on an appropriate test form.

**G. SAFETY AND HEALTH**

This method may involve hazardous materials, operations, and equipment. This method does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Prior to handling, testing or disposing of any of waste materials, testers are required to read: Part A (Section 5.0), Part B (Sections: 5.0, 6.0, 10.0 and 12.0) and Part C (Section 1.0) of Caltrans Laboratory Safety Manual. These sections pertain to requirements for general safety principles, standard operating procedures, protective apparel, disposal of materials and how to handle spills, accidents, emergencies, etc. Users of this method do so at their own risk.

**REFERENCE:**  
California Test 416

End of Text (California Test 416 contains 2 pages)

